FABRICATION AND CHARACTERIZATIONS OF METAL OXIDE NANOFIBERS FOR ENERGY APPLICATIONS

Sara Alkhalaf and Ram K. Gupta

Department of Chemistry, Pittsburg State University

Abstract

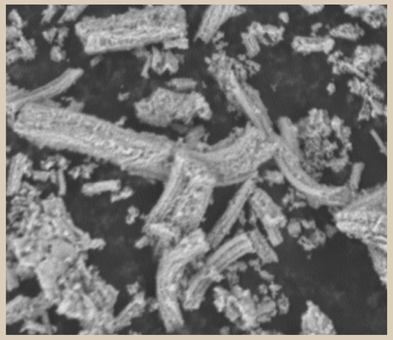
Nanostructured materials have attracted considerable research interest for their applications as catalyst, energy storage, fuel cells, etc. The main objective of this work is to synthesize and characterize nanofibers of metal oxides using electrospun technique and use them for energy storage applications. Various metal oxides such as $NiMn_2O_4$, $CoMn_2O_4$ and $ZnMn_2O_4$ were prepared as 1 dimensional (1-D) architecture using processable polymers and metal salts. The synthesized nanofibers were structurally and electrochemically characterized. The supercapacitive performance of these nanofibers was examined using cyclic voltammetry (CV) and galvanostatic charge-discharge techniques. The $CoMn_2O_4$ (CMO) nanofibers showed a promising value of ~ 120 F/g in 3M NaOH. The effect of different electrolytes such as LiOH, NaOH and KOH on the electrochemical properties of these metal oxide nanofibers was also investigated. It was observed that the charge storage capacity depends on the electrolyte used. The supercapacitor device fabricated using these nanofibers showed that charge storage capacity increases with increase in temperature. Our results suggest that electrospun nanofibers could be used for energy storage applications.

Experimental

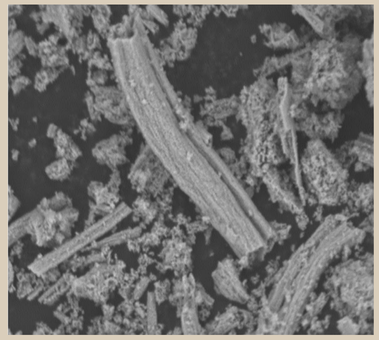
The material needed to synthesize the 1D nanofiber were 0.25 g of polyvinylpyrrolidone (PVP) , 0.25 g of polyacrylonitrile (PAN), 5 mL of N,N-dimethylformamide (DMF) , 0.5 mmol of $Co(CH_3COO)_2$ and 1 mmol of $Mn(CH_3COO)_2$. Making nanostructures requires going through three steps: The first step is making a homogeneous solution, the second step is elctrospinning, the last one is adjusting the heating rates in the furnace for each sample to get a different structure. To obtain a homogeneous solution, a mixture of 0.25 g of polyvinylpyrrolidone (PVP) and 0.25 g of polyacrylonitrile (PAN) were dissolved in 5 mL of N,N-dimethylformamide (DMF) solution, then that solution was stirred for one hour. After that 0.5 mmol of $Co(CH_3COO)_2$ and 1 mmol of $Mn(CH_3COO)_2$ were added. Afterward the solution was continuously stirred at room temperature for 24 hours.

After 24 hours the homogeneous solution was loaded into a plastic syringe with a stainless steel needle, then the syringe was connected to a high voltage power source. A high voltage of 18 kV was applied between the needle tip and aluminum foil collector with 16 cm space between the needle tip and the collector; the spinning rate is 1mL/h. The last step was putting the collected nanofibers into a calcinated furnace at 600 ° C for eight hours. To obtain a tube-in-tube structure the heating rate was 3 C/min. On the other hand the heating rate in a nanotube structure was 1 C/min and 5 C/min for solid 1D structure. The reason for that is when the heating rate is fast, the hard shells do not have enough time to form. The synthesis for the other nanofibers, which are $NiMn_2O_4$ and $ZnMn_2O_4$ was similar to the $CoMn_2O_4$.

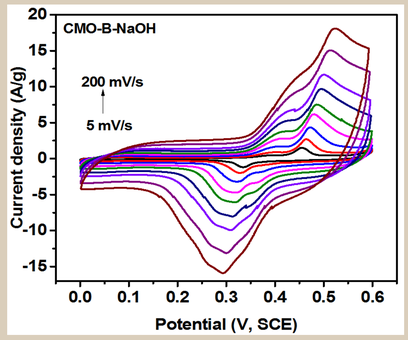
Results and Discussion



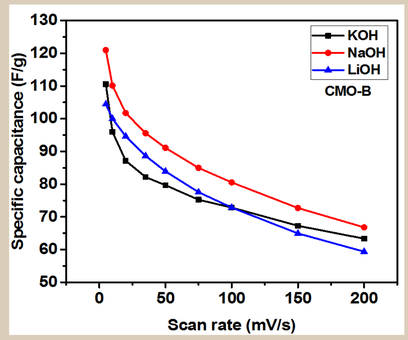
SEM picture for $ZnMn_2O_4$, tube-in-tube structure.



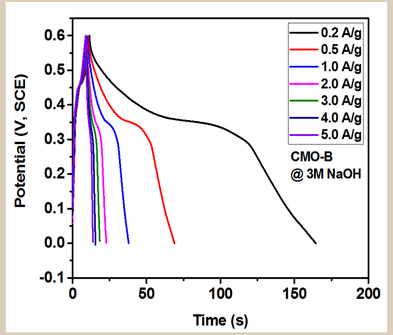
SEM picture for $ZnMn_2O_4$ 1D solid structure.



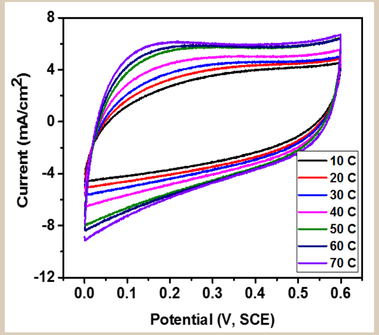
Cyclic voltammograms of CMO-B (heating rate 3 °C/min) sample at various scan rates in 3 M NaOH electrolyte.



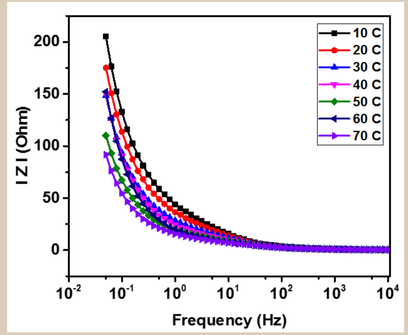
Variation of specific capacitance as a function of scan rate for CMO-B sample in different electrolytes.



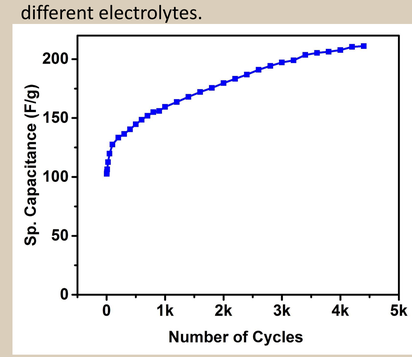
Galvanostatic charge-discharge characteristics of CMO-B electrode at various applied currents in 3M NaOH electrolyte



Cyclic voltammograms of the device at various temperatures.



Variation of impedance as a function of frequency and temperature.



Stability test for the CMO-B sample in 3M NaOH electrolyte

Summary

In summary, we have used an electrospinning technique to synthesize metal oxide nanofibers. It was observed that when the heating rate was changed, the structure and morphology of the nanofibers would be different. The electrochemical results suggest that $CoMn_2O_4$, $NiMn_2O_4$, and $ZnMn_2O_4$ nanofibers could be used as an electrode material for fabrication of high capacity supercapacitors. We studied the effect of temperature on the electrochemical behavior of the supercapacitor device. It was observed that temperature has a drastic effect on the charge storage capacity. The device showed about 75% improvement in charge storage capacity by increasing temperature from 10 to 70 °C.